PART III - SAMPLING AND ANALYSIS

8.0 SAMPLING

When testing is necessary, samples of dredged material, reference sediment, control sediment, organisms, and water will be needed for physical evaluations, chemical analysis, and for bioassay tests. This section provides general guidance for the development of a sampling plan including collection, handling and storage.

Sampling is the foundation upon which all testing rests but there are so many case-specific factors that influence sampling needs that detailed guidance of National scope is impractical. Some regions of the country have developed specific technical requirements and agency review/approvals of sampling and analysis plans. Regional guidance from local EPA and USACE offices should be sought for developing project-specific sampling plans as for information gathered at Tier I. The type of samples that may be required to complete the evaluations of Tiers II, III, and IV are outlined in Table 8-1. This manual provides general guidance on items of major importance to consider when designing a sampling plan. Additional guidance is provided by EPA (1995).

8.1 Preparation For Sampling

A well-designed sampling plan is essential when evaluating the potential impact of dredged material discharge upon the aquatic environment. Before any sampling is initiated, the sampling plan has to be tailored to meet clearly defined objectives for individual dredging operations. Factors such as the availability and content of historical data, the degree of sediment heterogeneity, the dredging depth, the number and geographical distribution of sample-collection sites, the procedures for collection, preservation, storage, and tracking of samples, and the necessity for adequate quality assurance and quality control (Appendix G; EPA, 1995) must be carefully considered. The magnitude of the dredging operation and its time and budgetary constraints should also be considered.

It is recommended that a written plan for sediment sampling and analyses be prepared and provided to the appropriate Federal and State agencies for coordination prior to sampling, where practicable. The Tier I evaluation would be a logical attachment to the sampling and analysis plan for agency review and comment. This coordination can reduce the chance of having to repeat costly procedures and can assist in keeping projects on schedule. An adequate amount of sediment and water should be collected to conduct planned evaluations and allow for any contingencies. Maximum allowable and recommended sample and organism holding times as well as the exigencies of resampling should be given careful consideration.

Table 8-1. Type of Samples Which May Be Required Following Tier I to Conduct Dredged-Material Evaluation Tests. Actual sampling requirements are project-specific and are determined during the development of the project plan. Sampling from the disposal site may also be conducted as necessary and appropriate, to verify the applicability of exclusion 230.60 (C) (see Sections 4.0 and 9.1.)

Tests	Water Samples			Sediment Samples			Biota Samples	
	Disposal Site	Dredging Site	Control[a]	Dredging Site	Reference Site	Control[a]	Dredging Site	Reference Site
Tier II								
Water column								
Screen	●[c]			●				
Elutriate	●[c]	●		●				
Tier II								
Benthic				●	●			
Tier III								
Water column	●[b]	●	●	●				
Tier III								
Benthic				●	●	●		
Tier IV								
Water column	●	●	●	●			●	●
Tier IV								
Benthic				●	●	●	●	●

[a]May or may not have to be field-collected.

[b]Dilution water for water column toxicity tests. Artificial or clean seawater or clean freshwater may also be used.

[c]Disposal site water is required for WQS comparison. Elutriate samples are prepared with dredging site water.

The importance of sampling is underscored by the fact that any evaluation is only as complete and reliable as the sampling (and sample handling and storage) upon which it is based. Thus, inadequacies or biases in sampling will limit the accuracy and/or the usefulness of the study results.

The primary objective of sediment and water collection is to obtain samples to adequately and accurately characterize the dredging and reference area. Sample size should be large enough to attain the appropriate detection limits but small enough to be conveniently handled and transported within the requirements for all planned analyses. The quality of the information obtained through the testing process is impacted by the following four factors:

- collecting representative samples
- collecting an appropriate number of samples
- using appropriate sampling techniques
- protecting or preserving the samples until they are tested.

Ideally, the importance of each of these three factors will be fully understood and appropriately implemented. In practice, however, this is not always the case. There may be occasions when study needs, time, costs or other resource constraints will limit the amount of information that should or can be gathered. When this is the case, the relative importance of each of these factors has to be carefully considered in light of the specific study purposes.

An important component of any field sampling program is a preproject meeting with all concerned personnel. Personnel involved may include management, field personnel, laboratory personnel, data management/analysis personnel, and representatives of regulatory agencies, the permit applicant, and the dredging company. To assure sampling quality, at least one individual familiar with the study area should be included in the preproject meeting. The purposes of the meeting include:

- defining the objectives of the sampling program
- ensuring communication among participating groups
- ensuring agreement on methods, QA/QC details and contingency plans.

The more explicitly the objectives of a testing program can be stated, the easier it will be to design an appropriate sampling plan. A complete sampling plan will result in a level of detail such that all sampling procedures and locations are clearly defined, sample volumes are clearly established, all logistical concerns are fully addressed, and target analytes are identified to class of compound.

8.2 Components Of A Sampling Plan

The following steps will help to ensure that all essential sampling plan information is provided:

- Review the plan for the proposed dredging operation, including the dimensions of the dredging area, the dredging depth(s), side-slopes, the volume of sediment for disposal, and the type of dredge equipment (e.g., clamshell, hydraulic) for determining composite sampling or delineating representative project segments.
- Evaluate the prior history and the existing database for the area, in particular, information gathered in Tier I. Identify relevant data and the need for additional data. Identify areas of potential environmental concern within the confines of the dredging operation.
- If appropriate, subdivide the dredging area into project segments on the basis of an assessment of level of environmental concern within the dredging area. This may be an iterative process that starts before sampling, using available information, and that is refined after sampling, based on new data.
- Determine the number of samples to be collected and select sampling locations. Choose methods and equipment for positioning vessels at established stations.
- Determine what sampling methods will be used.
- Define procedures for sample handling, preservation, storage, and (if applicable) field or shipboard analysis.
- Identify logistical considerations and safety precautions.

The subsections that follow discuss each of these steps and provide general guidance for their conduct. An essential step, preparation of a quality assurance/quality control (QA/QC) project plan, is discussed in detail in Appendix G and EPA (1995) and must be integral to the project. The QA/QC plan is essential to ensure that there will be sufficient and appropriate data of known and documented quality to make decisions with confidence and to defend those decisions. Properly prepared, a QA/QC plan expedites project coordination.

8.2.1 Review of Dredging Plan

A review of the plan for the dredging operation provides a basis for determining the sampling strategy. The volume of material to be dredged and the method of dredging are two important factors which will help to determine the number of samples required. The number of samples required is generally a judgement which considers the cost, resolution, and the risk of an incorrect decision regarding the volume of material to be dredged. Knowledge of the depth and physical characteristics of the material to be

dredged will help to determine the kind of sampling equipment that is required. The boundaries of the dredging area have to be known to ensure that the number and location of samples are appropriate. Sampling should generally be to the project depth (including overdredging) unless the sediments are known to be vertically homogeneous.

8.2.2 Historical Data

All information relevant to the dredging site should be reviewed. Using pertinent available information to determine project segments and station locations within the dredging area is both cost and technically effective. If a review of historical data identifies possible sources of contamination, skewing the sampling effort toward these areas may be justified for thorough characterization of these areas, but can lead to an incomplete assessment of contamination in the whole area. In areas of unequally distributed contamination, the total sampling effort should be increased to ensure representative, but not necessarily equal, sampling of the entire site. Sediment sampling techniques are detailed in Mudroch and MacKnight (1991). The information gathered for the Tier I evaluation (discussed in Section 4.1) should be reviewed for assistance in designing the sampling plan, in particular the following:

- **Geotechnical and hydrodynamic data**
  The grain size, specific gravity, water or solids content, total organic carbon (TOC) and identification of sediment horizons are helpful in making operational decisions. Areas of high currents and high wave energy tend to have larger grain-sized sediments than do quieter areas. Many contaminants have a greater affinity for clay and silt than for sand. Horizontal and vertical gradients may exist within the sediment. Local groundwater quality and movement should be determined if groundwater is a potential source of contamination.

- **Quality and age of available data**
  The value of the available data should be critically weighed. Existing high-quality data might lower costs by reducing the number of analytes measured or tests required for the proposed dredging operation. Existing data that do not meet all quality assurance/quality control (QA/QC) standards may still be useful if appropriate calibration and documentation are available; they are less useful if older methods with higher detection limits were used. Information from such studies might be helpful in identifying areas of contamination, but not in accurately assessing the degree of contamination.

- **Known distribution of contaminants**
  All evidence regarding contaminants within or near the dredging area, including spill data, may be an important consideration in identifying locations for sampling and/or determining sampling intensity.

- **Dredging history**
  Knowledge of prior dredging may dramatically affect sampling plans. If the area is frequently dredged (every 1-2 years) or if the sediments are subject to frequent mixing by wave action, currents, or ship traffic, the sediments are likely to be relatively homogeneous. Assuming that there is no major contaminant input, the sampling effort may be minimal. However, if there is information regarding possible contamination or heterogeneity is possible, a more extensive sampling effort may be indicated. New excavations of material unaffected by anthropogenic input may require less intensive sampling than maintenance dredging.

8.2.3 Subdivision of Dredging Area

Sediment characteristics are likely to vary substantially within the limits of the area to be dredged as a result of geographical and hydrological features. Areas of low hydrodynamic energy will be characterized by fine sediments that have a greater tendency to accumulate contaminants than do coarser-grained sediments. (However note that contaminants, if present in coarse-grained sediments, may be more bioavailable than if present in fine-grained sediments). Sediments in and downstream of heavily urbanized or industrialized areas are more likely to accumulate contaminants than sediments farther removed from direct contaminant input.

Many dredging operations can be subdivided into project segments (horizontal and/or vertical) which can be treated as separate management units. A project segment is an area expected to have relatively consistent characteristics that differ substantially from the characteristics of adjacent segments. Project segments may be sampled with various intensities and, if warranted by the study objectives and test results, the dredged material from various project segments can be managed differently during dredging and disposal to limit environmental impact. When the sampling plan is developed, project segments can be designated, based on factors including but not limited to: historical data, sediment characteristics, geographical configuration, anticipated method of dredging, depth of cut, sampling- or dredging-equipment limitations, results of pilot studies, and known or suspected contaminant concentrations. Surface sediments might be considered separately from subsurface sediments at the same location if vertical stratification of contamination is expected or encountered. Large dredging operations located

within industrialized areas might require subdivision into several project segments horizontally and into one or more segments vertically. A dredging operation characterized by relatively uniform distribution of sediment type in a nonindustrialized location might be considered as a single project segment. Vertical subdivisions usually are not appropriate in areas of rapid shoaling or in areas of high sediment mixing by ship scour, which are likely to be relatively homogenous vertically. Vertical subdivisions smaller than about 1 m are usually impractical because dredge operators generally cannot reliably control excavation with any finer precision; vertical subdivisions should reflect the actual removal precision to be employed during the dredging operation. If analytical data and test results for two or more project segments prove to be similar, these segments may be treated as one larger segment when considering disposal options. If the analytical and test results demonstrate important differences between project segments, alternative disposal options may be necessary for portions of the total sediment volume.

Any established sampling program should be sufficiently flexible to allow changes based on field observations; however, any deviations from the sampling plan must be documented, along with the rationale for such deviations. Certain characteristics of the sediments, such as color or texture, can be an indication of patchiness. The greater the patchiness, the larger the number of samples that will be required to adequately characterize the area. The project manager can refine a sampling program based on historical data and/or a preliminary sampling survey of the dredging area.

8.2.4 Selection of Sampling Locations and Number of Samples

Generally a single sampling strategy will be adequate for most circumstances. However, in some cases, two sampling strategies may be required. For instance, when sampling involves both uncontaminated and highly contaminated sediments with interfaces between the two, a single sampling strategy may not be sufficient to adequately characterize these sediments, which will probably be treated differently.

The method of dredging, the volume of sediment to be removed, the areal extent of the dredging project, and the horizontal and vertical heterogeneity of the sediment are key to determining station locations and the number of samples to be collected for the total dredging operation and for each project segment. When appropriate to testing objectives, samples may be composited prior to analysis (with attention to the discussion later in this section). The appropriate number of samples and the proper use of compositing should be determined for each operation on a case-by-case basis. Note that the following detailed discussion is not appropriate to all dredging operations. Sampling a number of small, isolated shoals is very different than sampling a large, contiguous open area.

Factors to Consider:

The following factors, many of which follow from information gathered in Tier I, should be among those considered in sampling station and pattern selection:

- objectives of the testing program
- bathymetry
- area of the dredging project
- accessibility
- flows (currents, tides)
- mixing (hydrology)
- sediment heterogeneity
- contaminant source locations
- land use activities
- available resources
- other physical characteristics.

Station Locations:

Station locations within the dredging area should include locations downstream from major point sources and in quiescent areas, such as turning basins, side channels, and inside channel bends, where fine-grained sediments are most likely to settle. Characteristics which help to define the representativeness of station(s) within a segment include:

- The distribution of sediments to be dredged is clearly defined.
- The project segment being sampled is clearly defined.
- The sampling locations are distributed appropriately within each project segment.
- Multiple samples should be collected if sample variability is suspected.
- When sediment variability is unknown, it may be necessary to conduct a preliminary survey of the dredging area to better define the final sampling program.

Sample Replication:

Within a station, samples may be collected for replicate testing. For this manual, laboratory replicates are generally recommended as opposed to field replicates, depending on site-specific issues. The former (subsamples of a composite sample of the replicates) involves pseudo-replication compared to separate samples for each replicate, but is more appropriate for dredged material evaluations where sediments will

be homogenized by the dredging and discharge process. The latter involves true replication but is more appropriate for field investigations of the extent and degree (or not) of homogeneity of sediment toxicity.

Depth Considerations:

Sediment composition can vary vertically as well as horizontally. Samples should be collected over the entire dredging depth (including over-dredging), unless the sediments are known to be vertically homogeneous or there are adequate data to demonstrate that contamination does not extend throughout the depth to be excavated. Separate analyses of defined sediment horizons may be useful to determine the vertical distribution of contamination if warranted by the study objectives. A major consideration of vertical compositing is the anticipated depth of dredging. For example, even though sediments in a 1 m shoal may vary in composition, the material would be mixed as a result of the dredging process.

Sampling Bias:

Ideally, the composition of an area and the composition of the samples obtained from that area will be the same. However, in practice, there often are differences due to bias in the sampling program, including disproportionate intensity of sampling in different parts of the dredging area and equipment limitations.

In some cases, to minimize bias, it may be useful to develop a sampling grid for each project segment. The horizontal dimensions of each project segment may be subdivided into grid cells of equal size, which are numbered sequentially. Cells are then selected for sampling either randomly or in an stratified random manner. It can be important to collect more than the minimum number of samples required, especially in areas suspected of having high or highly variable contamination. In some cases, although additional costs and logistic considerations will apply, extra samples may be archived (for long time periods in the case of physical characterization or chemical analyses and for short time periods in the case of biological tests), should reexamination of particular project segment(s) be warranted.

In other cases, a sampling grid may not be desirable. This is particularly the case where dredging sites are not continuous open areas, but are rather a series of separate humps, bumps, reaches and pockets with varying depths and surface areas. In these latter cases, sample distribution is commonly biased with intent.

Level of Effort:

In some cases, it may be advisable to consider varying the level of sampling effort. Project segments suspected or known to be contaminated may be targeted for an increased level of effort so that the

boundaries and characteristics of the contamination can be identified. A weighting approach can be applied whereby project segments are ranked in increasing order of concern, and level of concern can then be used as a factor when determining the number of samples within each project segment relative to other project segments.

Number of Samples:

In general, the number of samples that should be collected within each project segment is inversely proportional to the amount of known information, and is proportional to the level of confidence that is desired in the results and the suspected level of contamination. No specific guidance can be provided, but the following factors should be considered:

- the greater the number of samples collected, the better the areal and/or vertical definition
- single measurements are inadequate to describe variability
- the means of several measurements at each station within a project segment generally are less variable than individual measurements at each station.

Time and Funding Constraints:

In all cases, the ultimate objective is to obtain sufficient information to evaluate the environmental impact of a dredged material disposal operation. The realities of time and funding constraints have to be recognized, although such do not justify inadequate environmental evaluation. Possible responses to cost constraints have been discussed by Higgins (1988). If the original sampling design does not seem to fit time or funding constraints, several options are available, all of which increase the risk of an incorrect determination:

- Reduce the number of project segments into which the project is divided, but maintain the same total number of samples.
- Maintain (or even increase) the number of stations sampled, and composite multiple samples from within a project segment so that a lower number of analyses are performed per project segment.

Project Segments:

Regardless of the final decision on project segments and the number of sample stations and replicates per project segment, expected or known degree of contamination will be the dominant factor in initially describing the proposed project segments. If variation in potential dredged material impact within a project segment is likely, where possible it may be advisable either to use a stratified random-sampling approach or to redefine project-segment boundaries. Once sampling data are available, it is advisable to reconsider the boundaries of the project segments to be used in the actual dredging in order to maximize homogeneity within segments.

Sample Compositing:

The objective of obtaining an accurate representation and definition of the dredging area and method has to be satisfied when compositing samples. Compositing provides a way to control cost while still analyzing sediments from a large number of stations. Compositing results in a less detailed description of variability within the area sampled than would individual analysis at each station. However if, for example, five analyses can be performed to characterize a project segment, the increased coverage afforded by collecting 15 individual samples and combining sets of three into five composite samples for analysis may justify the increased time and cost of collecting the extra 10 samples. Compositing can also provide the large sample volumes required for some biological tests. Composite samples represent the "average" of the characteristics of the individual samples making up the composite and are generally appropriate for logistic and other reasons; however, composite samples which serve to "dilute" a highly toxic but localized sediment "hot spot" are not recommended. Further, composite samples are not recommended for stations with very different sediment grain size characteristics.

Sample Definition:

When a sediment sample is collected, a decision has to be made as to whether the entire sediment volume is to be considered as the sample or whether the sediment volume represents separate samples. For instance, based on observed stratification, the top 1 m of a core might be considered to be a separate sample from the remainder of the core. After the sediment to be considered as a sample is identified, it should be thoroughly homogenized. Samples may be split before compositing, with a portion of the original sediment archived for possible later analysis, and the remainder combined with parts of other samples. These are then thoroughly homogenized (using clean instruments until color and textural homogeneity are achieved), producing the composite sample.

8.2.5 Sample Collection Methods

Sample collection requires an adequately trained crew, an adequate vessel equipped with navigational and supporting equipment appropriate to the site and the study, and noncontaminating sampling apparatus capable of obtaining representative samples. Divers may also be used in some cases to collect some samples; in such cases divers must be certified and approved diver safety management plans must be in place. To assure sampling quality, at least one individual familiar with the study area should be present during the sampling activities. Sampling effort for a proposed dredging operation is primarily oriented toward collection of sediment samples for physical and chemical characterization and for biological tests. Collection of water samples is also required to evaluate potential water column impact. Collection of organisms near the disposal site might be necessary if there is a need to characterize indigenous populations or to assess concentrations of contaminants in tissues. Organisms for use in toxicity and bioaccumulation tests may also be field-collected.

In general, a hierarchy for sample collection should be established to prevent contamination from the previous sample, especially when using the same sampling apparatus to collect samples for different analyses. Where possible, the known, or expected, least contaminated stations should be sampled first. At a station where water and sediment are to be collected, water samples should be collected prior to sediment samples. The vessel should ideally be positioned downwind or downcurrent of the sampling device. When raising or lowering sampling devices, care should be taken to avoid visible surface slicks and the vessel's exhaust. The deck and sample handling area should be kept clean to help reduce the possibility of contamination.

8.2.5.1 Sediment Sample Collection

Mudroch and MacKnight (1991) provide useful reference information. Higgins and Lee (1987) provide a perspective on sediment collection and analysis as commonly practiced in USACE Districts. ASTM (1994a) and Burton (1991) provide guidelines for collecting sediments for toxicological testing. Guidance provided in these publications may be followed on all points that do not conflict with this manual.

Care should be taken to avoid contamination of sediment samples during collection and handling. A detailed procedure for handling sampling equipment and sample containers should be clearly stated in the sampling plan associated with a specific project. This may be accomplished by using standard operating procedures (SOPs). For example, samples designated for trace metal analysis should not come into contact with metal surfaces (except stainless steel, unless specifically prohibited for a project), and samples designated for organic analysis should not come into contact with plastic surfaces. Samples for

biological tests may be stored in clean polypropylene containers. Subsamples for particular groups of analytes may be removed from areas of the sample not in physical contact with the collecting instrument.

A coring device with appropriate liners is recommended whenever sampling to depth is required. The choice of corer design depends upon factors including the objectives of the sampling program, sediment volumes required for testing, sediment type, water depth, sediment depth, and currents or tides. A gravity corer may be limited to cores of 1-2 m in depth, depending upon sediment grain size, degree of sediment compactness, and velocity of the drop. For penetration greater than 2 m, a vibratory corer or a piston corer is generally preferable. These types of coring devices are generally limited to soft, unconsolidated sediments. A split-spoon core may be used for more compacted sediment. The length of core that can be collected is usually limited to 10 core diameters in sand substrate and 20 core diameters in clay substrate. Longer cores can be obtained, but substantial sample disturbance results from internal friction between the sample and the core liner.

Freefall cores can cause compaction of the vertical structure of sediment samples. Therefore, if the vertical stratification in a core sample is of interest, a piston or vibra corer should be used. Piston corers use both gravity and hydrostatic pressure. As the cutting edge penetrates the sediments, an internal piston remains at the level of the sediment/water interface, preventing sediment compression and overcoming internal friction. A vibra corer is a more complex piece of equipment but is capable of obtaining 3- to 7-m cores in a wide range of sediment types by vibrating a large diameter core barrel through the sediment column with little compaction. If the samples will not be sectioned prior to analysis, compaction is not a problem, and noncontaminating freefall corers are a suitable alternative.

Corers are the samplers of preference in most cases because of the variation in contamination with depth that can occur in sediment deposits. Substantial variation with depth is less likely in shallow channel areas without major direct contaminant inputs, that have frequent ship traffic, and from which sediments are dredged at short intervals. Generally, in these situations, accumulating sediments are resuspended and mixed semicontinuously by ship scour and turbulence, effectively preventing stratification. In such cases, surface grab samples can be representative of the mixed sediment column, and corers should be necessary only if excavation of infrequently disturbed sediments below the mixed layer is planned.

Grab samplers are also appropriate for collecting surficial samples of reference or control sediments. A grab can be Teflon-coated to prevent potential contamination of trace metal samples. The sampling device should at least be rinsed with clean water between samples and possibly also solvent-rinsed.

8.2.5.2 Water Sample Collection

If water samples are necessary, representative samples should be collected with either a noncontaminating pump or a discrete water sampler. When sampling with a pump, the potential for contamination can be minimized by using a peristaltic or a magnetically coupled impeller-design pump. The system should be flushed with the equivalent of 10 times the volume of the collection tubing. Also, any components within several meters of the sample intake should be noncontaminating (i.e., sheathed in polypropylene or epoxy-coated). Potential sample contamination must be avoided, including vessel emissions and other sampling apparatus.

A discrete water sampler should be of the close/open/close type so that only the target water sample comes into contact with internal sampler surfaces. Seals should be Teflon-coated whenever possible. Water sampling devices should be acid-rinsed (1:1 nitric acid) prior to use for collection of trace-metal samples, and solvent-rinsed prior to collection of samples for organic analyses.

8.2.5.3 Organism Collection

Benthic organism collection methods may be species specific and can include, but are not restricted to, bottom trawling, grabs or cores. If organisms are to be maintained alive, they should be transferred immediately to containers with clean, well-oxygenated water, and sediment as appropriate. Care must be taken to prevent organisms from coming into contact with potentially contaminated areas or fuels, oils, natural rubber, trace metals, or other contaminants.

8.2.6 Sample Handling, Preservation, and Storage

Detailed procedures for sample handling, preservation, and storage should be part of the standard operating procedures and protocols developed for each sampling operation. Samples are subject to chemical, biological, and physical changes as soon as they are collected. Sample handling, preservation, and storage techniques have to be designed to minimize any changes in composition of the sample by retarding chemical and/or biological activity and by avoiding contamination. Collection methods, volume requirements, container specifications, preservation techniques, storage conditions and holding times (from the time of sample collection) for sediment, water, and tissue samples are discussed below and summarized in Table 8-2.

8.2.6.1 Sample Handling

Sufficient sample volume must be collected to:

Table 8-2. Summary of Recommended Procedures for Sample Collection, Preservation, and Storage.[a]

Analyses	Collection Method[b]	Amount Required[c]	Container[d]	Preservation Technique	Storage Conditions	Holding times[e]
SEDIMENT						
Chemical/Physical Analyses						
Metals	Grab/corer	100 g	Precleaned polyethylene jar[f]	Dry ice[f] or freezer storage for extended storages; otherwise refrigerate	≤ 4°C	Hg - 28 days Others - 6 months[g]
Organic compounds (e.g., PCBs, pesticides, polycyclic aromatic hydrocarbons)	Grab/corer	250 g	Solvent-rinsed glass jar with Teflon lid[f]	Dry ice[f] or freezer storage for extended storages; otherwise refrigerate	≤ 4°C[f]/dark[g]	14 days[h]
Particle size	Grab/corer	100 g	Whirl-pac bag[f]	Refrigerate	< 4°C	Undetermined
Total Organic Carbon (TOC)	Grab/corer	50 g	Heat treated glass vial with Teflon-lined lid[f]	Dry ice[f] or freezer storage for extended storages; otherwise refrigerate	≤ 4°C[f]	14 days
Total solids/ specific gravity	Grab/corer	50 g	Whirl-pac bag	Refrigerate	< 4°C	Undetermined
Miscellaneous	Grab/corer	≥ 50g	Whirl-pac bag	Refrigerate	< 4°C	Undetermined

Table 8-2 *(continued)*

Analyses	Collection Method[b]	Amount Required[c]	Container[d]	Preservation Technique	Storage Conditions	Holding times[e]
SEDIMENT (continued)						
Sediment from which elutriate is prepared	Grab/corer	Depends on tests being performed	Glass with Teflon-lined lid	Completely fill and refrigerate	4°C/dark/airtight	14 days
Biological Tests						
Dredged material	Grab/corer	12-15 L per sample	Plastic bag or container[i]	Completely fill and refrigerate; sieve	4°C/dark/airtight	14 days[j]
Reference sediment	Grab/corer	45-50 L per test	Plastic bag or container[i]	Completely fill and refrigerate; sieve	4°C/dark/airtight	14 days[j]
Control Sediment	Grab/corer	21-25 L per test	Plastic bag or container[i]	Completely fill and refrigerate; sieve	4°C/dark/airtight	14 days[j]
WATER AND ELUTRIATE						
Chemical/Physical Analyses						
Particulate analysis	Discrete sampler or pump	500 - 2000 mL	Plastic or glass	Lugols solution and refrigerate	4°C	Undetermined
Metals	Discrete sampler or pump	1 L	Acid-rinsed polyethylene or glass jar[k]	pH <2 with HNO_3[k]; refrigerate	4°C 2°C[k]	Hg - 14 days Others - 6 months[l]
Total Kjeldahl nitrogen (TKN)	Discrete sampler or pump	100 - 200 mL	Plastic or glass[l]	H_2SO_4 to pH <2; refrigerate	4°C[l]	24 h[l]
Chemical oxygen demand (COD)	Discrete sampler or pump	200 mL	Plastic or glass[l]	H_2SO_4 to pH <2; refrigerate	4°C[l]	7 days[l]

Table 8-2 *(continued)*

Analyses	Collection Method[b]	Amount Required[c]	Container[d]	Preservation Technique	Storage Conditions	Holding times[e]
WATER AND ELUTRIATE (continued)						
Total organic carbon (TOC)	Discrete sampler or pump	100 mL	Plastic or glass[l]	H_2SO_4 to pH <2; refrigerate	4°C[l]	<48 h[l]
Total inorganic carbon (TIC)	Discrete sampler or pump	100 mL	Plastic or glass[l]	Airtight seal; refrigerate[h]	4°C[l]	6 months[l]
Phenolic compounds	Discrete sampler or pump	1 L	Glass[l]	0.1 - 1.0 g $CuSO_4$; H_2SO_4 to pH <2; refrigerate	4°C[l]	24 h[l]
Soluble reactive phosphates	Discrete sampler or pump	-	Plastic or glass[l]	Filter; refrigerate[h]	4°C[l]	24 h[l]
Extractable organic compounds (e.g., semivolatiles)	Discrete sampler or pump	4 L	Amber glass bottle[k]	pH < 2, 6N HCl; airtight seal; refrigerate	4°C[k]	7 days for extraction; 40 days for extract analysis[k]
Volatile organic compounds	Discrete sampler or pump	80 mL	Glass vial[k]	pH < 2 with 1:1 HCL; refrigerate in airtight, completely filled container[k]	4°C[k]	14 days for sample analysis if preserved[m]
Total phosphorus	Discrete sampler or pump	-	Plastic or glass[i]	H_2SO_4 to pH < 2; refrigerate	4°C[l]	7 days[l]
Total solids	Discrete sampler or pump	200 mL	Plastic or glass[l]	Refrigerate	4°C[l]	7 days[l]

Table 8-2 *(continued)*

Analyses	Collection Method[b]	Amount Required[c]	Container[d]	Preservation Technique	Storage Conditions	Holding times[e]
WATER AND ELUTRIATE (continued)						
Volatile solids	Discrete sampler or pump	200 mL	Plastic or glass[l]	Refrigerate	4°C[l]	7 days[l]
Sulfides	Discrete sampler or pump	-	Plastic or glass[l]	pH > 9 NaOH (ZnAc); refrigerate	4°C[l]	24 h[l]
Biological Tests						
Site water	Grab	Depends on tests being performed	Plastic carboy	Refrigerate	< 4°C	14 days
Dilution water	Grab or makeup	Depends on tests being performed	Plastic carboy	Refrigerate	<4°C	14 days
TISSUE						
Metals	Trawl/Teflon-coated grab	5-10 g	Double Ziploc[f]	Handle with nonmetallic forceps; plastic gloves; dry ice[f]	≤ -20°C[f] or freezer storage	Hg - 28 days Others - 6 months[n]
PCBs and chlorinated pesticides	Trawl/Teflon-coated grab	10-25 g	Hexane-rinsed double aluminum foil and double Ziploc[f]	Handle with hexane-rinsed stainless steel forceps; dry ice[f]	≤ -20°C[f] or freezer storage	14 days[h]
Volatile organic compounds	Trawl/Teflon-coated grab	10-25 g	Heat-cleaned aluminum foil and watertight plastic bag[m]	Covered ice chest[g]	≤ -20°C[h] or freezer storage	14 days[n]

Table 8-2 *(continued)*

Analyses	Collection Method[b]	Amount Required[c]	Container[d]	Preservation Technique	Storage Conditions	Holding times[e]
TISSUE (continued)						
Semivolatile organic compounds (e.g, PAH)	Trawl/Teflon-coated grab	10-25 g	Hexane-rinsed double aluminum foil and double Ziploc[f]	Handle with hexane-rinsed stainless steel forceps; dry ice[f]	≤ -20°C[f] or freezer storage	14 days[h]
Lipids	Trawl/Teflon-coated grab	part of organic analyses	Hexane-rinsed aluminum foil	Handle with hexane-rinsed stainless steel forceps; quick freeze	≤ -20°C or freezer storage	14 days[h]

[a] This table contains only a summary of collection, preservation, and storage procedures for samples. The cited references should be consulted for a more detailed description of these procedures.

[b] Collection method should include appropriate liners.

[c] Amount of sample required by the laboratory to perform the analysis (wet weight or volume provided, as appropriate). Miscellaneous sample size for sediment should be increased if auxiliary analytes that cannot be included as part of the organic or metal analyses are added to the list. The amounts shown are not intended as firm values; more or less tissue may be required depending on the analytes, matrices, detection limits and particular analytical laboratory.

[d] All containers should be certified as clean according to EPA (1990a).

[e] These holding times are for sediment, water, and tissue based on guidance that is sometimes administrative rather than technical in nature. There are no promulgated, scientifically based holding time criteria for sediments, tissues or elutriates. References should be consulted if holding times for sample extracts are desired. Holding times are from the time of sample collection.

[f] NOAA (1989)

[g] Tetra Tech (1986a)

[h] Sample may be held for up to one year if ≤ -20°C.

[i] Polypropylene should be used if phthalate bioaccumulation is of concern.

[j] Two weeks is recommended; sediments must not be held for longer than 8 weeks prior to biological testing.

[k] EPA (1987c); 40 CFR Part 136, Table III

[l] Plumb (1981)

[m] If samples are not preserved to pH<2, then aromatic compounds must be analyzed within 7 days.

[n] Tetra Tech (1986b)

- perform the necessary analyses
- partition the samples, either in the field or as soon as possible after sampling, for respective storage and/or analytical requirements (e.g., freezing for trace metal analysis, refrigeration for bioassays)
- provide sample for replicate or QA analyses, if specified
- archive portions of the sample for possible later analysis.

Sample handling is project and analysis specific as well as being based on what is practical and possible. Generally, samples to be analyzed for trace metals should not come into contact with metals, and samples to be analyzed for organic compounds should not come into contact with plastics. All sample containers should be appropriately cleaned (acid-rinsed for analysis of metals; solvent-rinsed for analysis of organic compounds).

For analysis of volatile compounds, samples should completely fill the storage container, leaving no air-space. These samples should be refrigerated but never frozen or the containers will crack. Samples for other kinds of chemical analysis are sometimes frozen. If the sample is to be frozen, sufficient air space should be allowed for expansion to take place. Container labels have to withstand soaking, drying, and freezing without becoming detached or illegible. The labelling system should be tested prior to use in the field.

Sediment samples for biological testing should have at least the larger living organisms removed from the sediment prior to testing. This may be accomplished by press-sieving the sediments through a 1-mm-mesh screen. Other matter retained on the screen with the organisms, such as shell fragments, gravel, and debris, should be recorded and discarded. Prior to use in bioassays, individual test sediments should be thoroughly homogenized with clean instruments (until color and textural homogeneity is achieved).

8.2.6.2 Sample Preservation

Preservation steps should be taken immediately upon sediment collection. There is no universal preservation or storage technique although storage in the dark at 4°C is generally used for all samples held for any length of time prior to partitioning, and for some samples after partitioning. A technique for one group of analyses may interfere with other analyses. This problem can be overcome by collecting sufficient sample volume to utilize specific preservation or storage techniques for specific analytes or

tests. Preservation, whether by refrigeration, freezing, or addition of chemicals, should be accomplished onboard the collecting vessel whenever possible. If final preservation techniques cannot be implemented in the field, the sample should be temporarily preserved in a manner that retains its integrity.

Onboard refrigeration is generally accomplished with coolers and ice; however, samples should be segregated from melting ice or cooling water. Samples which are to be frozen on board may be stored in an onboard freezer or may simply be placed in a cooler with dry ice or blue ice. Sediment samples for biological analysis should be preserved at 4°C, never frozen or dried. Additional guidance on sample preservation is given in Table 8-2.

8.2.6.3 Sample Storage

The elapsed time between sample collection and analysis should be as short as possible. Sample holding times for chemical evaluations are analysis-specific (Table 8-2). Sediments for bioassay (toxicity and/or bioaccumulation) testing *should* be tested as soon as possible, preferably within 2 weeks of collection. Studies to date suggest that sediment storage time should not exceed 8 weeks (at 4°C, in the dark, excluding air) (Becker and Ginn, 1990; Tatem et al., 1991). Toxicity may change with storage time. Sample storage conditions (e.g., temperature, location of samples) should be documented.

8.2.7 Logistical Considerations and Safety Precautions

A number of frustrations in sample collection and handling can be minimized by carefully thinking through the process and requirements before going to the field (e.g., see EPA, 1995). Contingency plans are essential. Well-trained, qualified, and experienced field crews should be used. Backup equipment and sampling gear, and appropriate repair parts, are advisable. A surplus of sampling containers and field data sheets should be available. Sufficient ice and adequate ice-chest capacity should be provided, and the necessity of replenishing ice before reaching the laboratory should be considered. A vessel with adequate deck space is safer and allows for more efficient work than an overcrowded vessel. Unforeseeable circumstances (e.g., weather delays) are to be expected during field sampling, and time to adequately accomodate the unforeseen has to be included in sampling schedules.

Appropriate safety and health precautions must be observed during field sampling activities. EPA (1984) should be used as a guidance document to prepare a site-specific health and safety plan. The health and safety plan should be prepared as a separate document from the QA project plan. Requirements set forth in the Occupational Safety and Health Administration 29 CFR § 1910.120 (Federal Register, Vol. 54, No. 43) should be met for medical surveillance, personal protection, respirator fit testing (if applicable),

and hazardous waste operations training (if applicable) by all personnel working in contaminated areas or working with contaminated media.

The procedures and practices established in the site-specific health and safety plan must be observed by all individuals participating in the field activities. Safety requirements should also be met by all observers present during field audits and inspections. The plan should include the following information:

- site location and history
- scope of work
- site control
- hazard assessment (chemical and physical hazards)
- levels of protection and required safety equipment
- field monitoring requirements
- decontamination
- training and medical monitoring requirements
- emergency planning and emergency contacts.

Samples must be properly disposed when no longer needed. Ordinary sample-disposal methods are usually acceptable, and special precautions are seldom appropriate. Under Federal law [40 CFR 261.5(a)], where highly contaminated wastes are involved, if the waste generated is less than 100 Kg per month, the generator is conditionally exempt as a small-quantity generator and may accumulate up to 1,000 Kg of waste on the property without being subject to the requirements of Federal hazardous waste regulations. However, State and local regulations may require special handling and disposal of contaminated samples. When samples have to be shipped, 49 CFR 100-177 should be consulted for current Department of Transportation regulations on packing and shipping.

8.2.8 Non-Indigenous Test Species

Over the last few years, there has been a growing awareness of the ecological and economic damage caused by introduced species. Because both east and west coast species are often used in bioaccumulation

tests, there is a real potential of introducing bioaccumulation test species or associated fauna and flora (e.g., pathogens, algae used in transporting the worms). It is the responsibility of the persons conducting the bioaccumulation or toxicity tests to assure that no non-indigenous species are released.

The general procedures to contain non-indigenous species are to collect and then poison all water, sediment, organisms and associated packing materials (e.g., algae, sediment) before disposal. Chlorine bleach can be used as the poison. A double containment system is used to keep any spillage from going down the drain. Guidance on procedures used in toxicity tests can be found in Appendix B of DeWitt et al. (1992a). Flow-through tests can generate large quantities of water, and researchers should plan on having sufficient storage facilities.